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Data Collection Protocols for Adhesive Testing Results Using the Materials Selection and Analysis Tool

by Daniel DeSchepper, David Flanagan, Jonathan Kaufman, Benjamin Henrie, Wendy Kosik Chaney, and Robert Jensen

ARL-RP-381 **June 2012**

A reprint from *SAMPE 2012,* Baltimore, MD, 21–24 May 2012.

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Army Research Laboratory
Aberdeen Proving Ground, MD 21005-5069

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The selection and substitution of materials is the keystone of successful engineering. Armor represents a complex and broad spectrum of possible designs that are continually evolving to meet the protection needs imposed by ever emerging threats. Adhesive selection plays a critical role in armor design. Hence, it is vital to capture, consolidate and organize adhesive data in a meaningful way for both engineering design as well as material advancement. A multitude of adhesives have been available from the commercial market over the years. Those intended for aerospace applications tend to have the highest pedigree engineering criteria defined within existing databases. The Army's adhesive needs push the quest for desirable properties well outside of the aerospace regime, which makes a trial and error selection approach both costly and time consuming. Materials informatics and data mining computational tools are now moving towards the practicality needed for drawing accurate correlations between complex high loading rate response and simpler quasi-static properties.

Through a DoD-NASA partnership, the Army Research Laboratory has developed an adhesive database using the ARL tailored Materials Selection and Analysis Tool (MSAT) platform. Leveraging MSAT's real-time data management platform, we have refined our experimental data collection methodology to capture maximum detail, data pedigree, and integrity of non-aerospace adhesives on a large scale. As non-aerospace adhesives represent the dominant sector of the commercial market, streamlining the testing and data collection strategy is critical in achieving a reliable database flexible enough to respond to ever-shifting Army driven property requirements. A key, and often underestimated, factor in the data collection is in the transference process from experimental testing results to digital format, which is focus of this current work.

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DATA COLLECTION PROTOCOLS FOR ADHESIVE TESTING RESULTS USING THE MATERIALS SELECTION AND ANALYSIS TOOL

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1. INTRODUCTION

1.1 Adhesively Bonded Armor

Composite integral armor (CIA) has been the subject of ongoing research as a candidate to replace traditional rolled homogeneous armor steel in some ground vehicle applications, primarily due to lower areal density and the demand for increased vehicle mobility and fuel efficiency.[1] In this type of armor assembly the incoming ballistic penetrator is defeated through an erosion mechanism in a ceramic strike face, which is supported by a fiber reinforced composite backing plate (or low density metal). The backing plate allows for maximum dwell time by maintaining a state of compression in the ceramic. As relatively straight-forward as this concept is, when theory is put to practice the enormous magnitude of complexity involving the analysis and interpretation of the response of an integrated materials system to impact and ballistic event remains a significant challenge.[2,3,4,5,6]

Furthermore, as the composition of CIA includes non-metallic materials, traditional welding techniques are not an assembly option, which is why secondary polymeric adhesives are used to bond the armor packages together. The failure modes of CIA represent an inter-related array (both in time and length scale) in which adhesive failure is particularly detrimental to both structural and ballistic performance. Regardless of the specific failure mode in any of the CIA backing materials behind the ceramic, once the ceramic becomes unsupported the loading due to the incoming projectile is biased from a stronger compressive to much weaker tensile mode and the primary defeat mechanism of erosion is negated. Adhesive failure represents a significant element of the global failure modes in CIA, yet very little understanding of the adhesive response during high loading rate events is known. To compound the difficulties, very little quantitative information exists for correlating basic quasi-static coupon level testing to empirically observed high loading rate testing results.

1.2 A Broad Range of Adhesives are Needed for Ground Vehicle Applications

Aerospace material property requirements are dominated by “strength and stiffness”, which are well defined for the commercial adhesive industry and can trace their roots to the 1930’s.[7,8,9,10] The correlations between aerospace materials properties with processing, performance, design, structure, and manufacturing have also been studied extensively.[11] Ground vehicle armor material property requirements are dominated by “strength and damage tolerance”, which have not been a well defined objective for the commercial adhesive industry.[12] The high loading rate environment of the armored ground vehicle and the expectations of receiving extensive combat damage without compromising mission capability are fundamentally different than aviation.[1] However, from qualitative observations, it is known that adhesives with high single-lap-joint bond strengths, high tensile strengths, and high strains to failure measured using standardized quasi-static testing protocols tend to show the higher damage tolerance traits needed for ground vehicle applications. Furthermore, as armor design is continuously evolving to match a very broad range of continually emerging threats from the field, there exists no, and will never exist, a single adhesive that is universally “the best for armor”. An Army derived performance requirement for ground vehicle armor applications should convey these empirical high loading rate observations, which will result in a standards document with a much broader acceptance

region than offered by contemporary examples from aviation, which is graphically estimated in Figure 1.

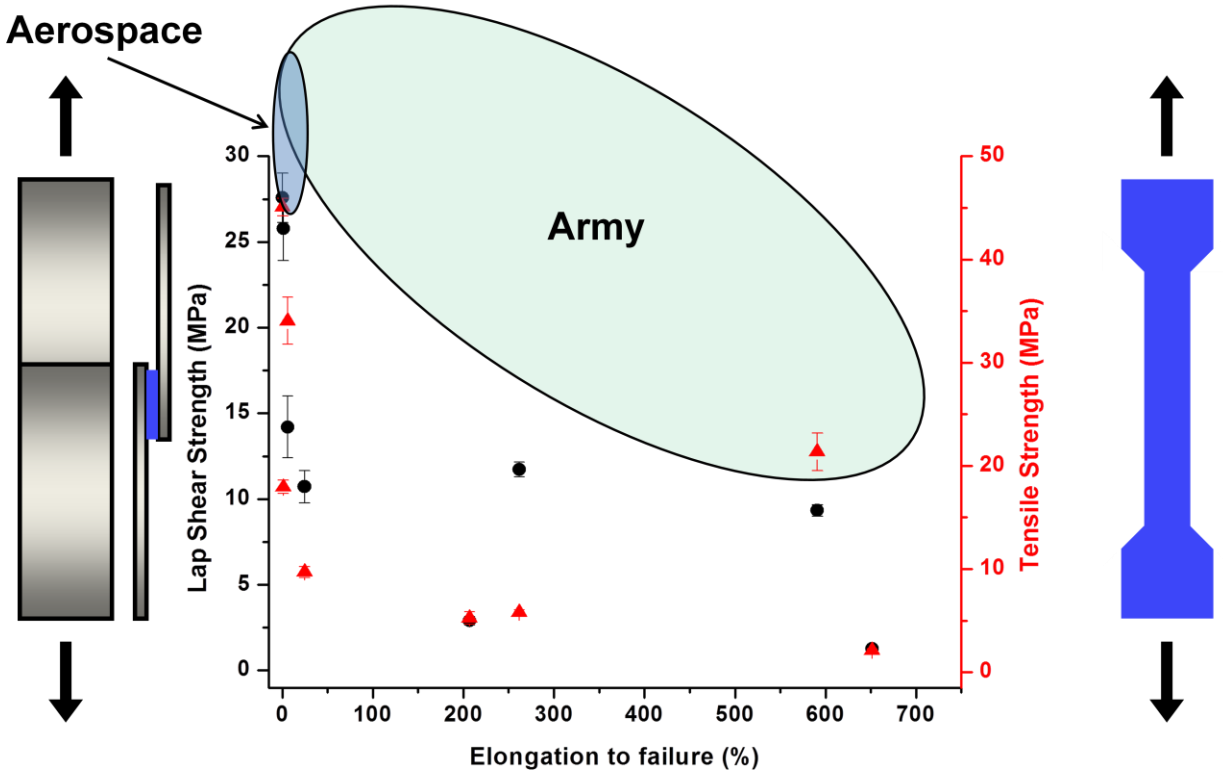

Figure 1. Adhesives with high single-lap-joint bond strengths, high tensile strengths, and high strains to failure measured using standardized quasi-static testing protocols tend to show the higher damage tolerance traits needed for ground vehicle applications. Results were measured at room temperature and quasi-static rates

Implications of a Broad Adhesives Need for Army Standardization

It is expected that Army driven adhesive strength requirements, based upon a very broad range of elongation to failure, will invoke a high level of chemical diversity. Such variety will be impossible to cover within a single adhesive chemical family. In addition to familiar adhesives derived from epoxies, phenolics, polyurethanes, polyureas, acrylics, silicones, and polyimides, it is possible that newer adhesives inspired by recent advances in biology or nano technology will need to be developed. It is also expected that the processing envelope will also be equally broad, as the Army employs thermosetting, thermoplastic, paste, and film adhesives cured using a variety of autoclave and out-of-autoclave bonding techniques.

While the conceptual Army requirements portrayed in Figure 1 are fairly straight-forward, the potential breadth of adhesive candidates will place a substantially increased need to accurately capture test sample processing ‘metadata’ along with the quantified testing results. In this paper

we use the term metadata to refer to any data which increases confidence in materials pedigree and the test specimen's provenance, to ensure data integrity. To show the importance of data integrity and accurate metadata we report the variability of adhesive single-lap-joint strengths obtained from a commercial epoxy film adhesive by varying the processing conditions. We also demonstrate the single-lap-joint adhesive bond performance of a polyurea paste adhesive, which resulted in significant non-linear behavior.

2. EXPERIMENTATION

2.1 Single-Lap-Joint Fabrication and Testing

ASTM D 1002[13] was the basis standard used for the single-lap-joint testing. Aluminum adherends (Alloy 2024-T3) were used with dimensions of 25.4 mm x 101.6 mm x 1.62 mm. The aluminum was machined using a template that allowed for the simultaneous bonding of 5 individual samples while controlling the bond thickness and alignment using a custom tooling fixture, as shown in Figure 2. The steel fixture secures the aluminum templates with a 12.7 mm bonded joint overlap using steel guide pins. A shim panel is used to set the bond thickness, which was set at 0.81 mm, except where noted. Once the panels have been adhered together the individual panels are cut apart. Single-Lap-Joint tests were performed using a 5500 series Instron testing machine in tension mode equipped with a 22 kN load cell and mechanical wedge grips. The length in the jaws of the grippers is set at 25.4 mm. The lap shear tests were run at a rate of 1.27 mm/min per the ASTM standard.

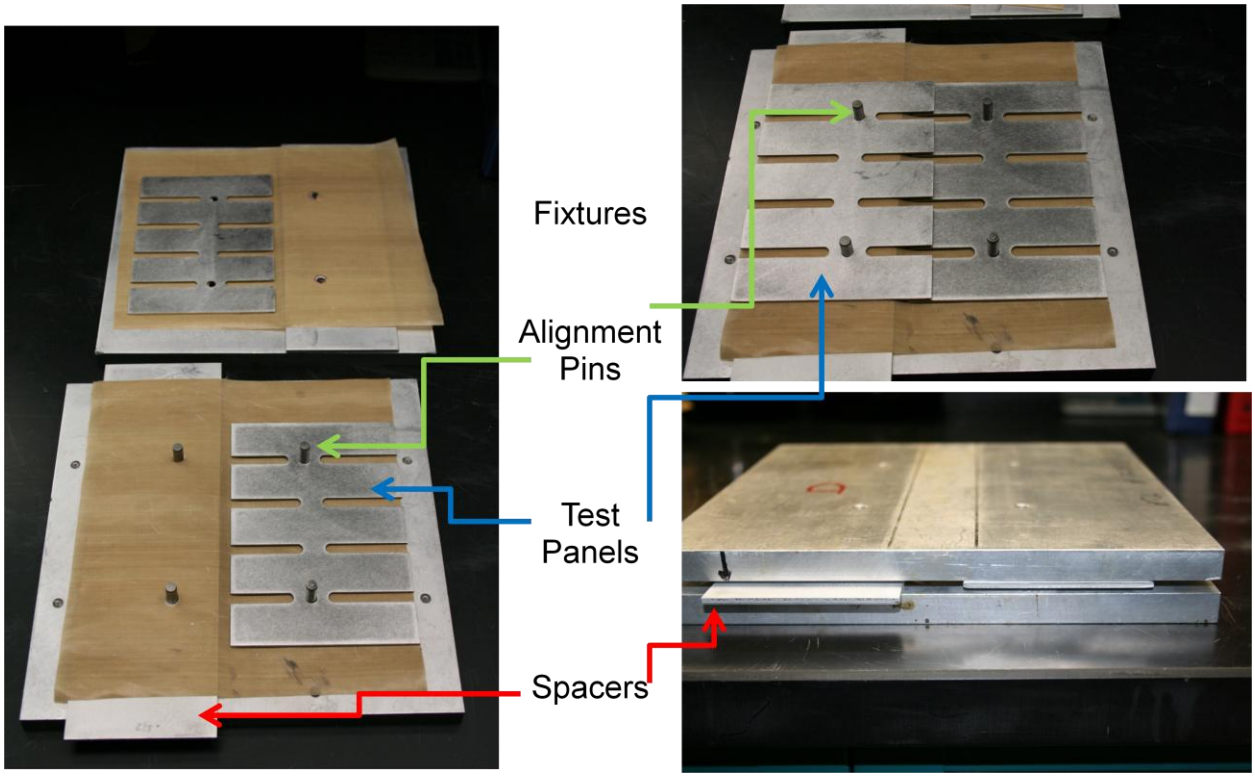

Figure 2. Custom tooling fixture and ASTM D1002 aluminum coupon templates prior to adhesive bonding. Individual single-lap-joints were cut from bonded coupon assembly after cure.

2.2 Surface Preparation

Grit Blast – Silane Treatment

Both sides of the aluminum lap-shear joint panels were wiped down with acetone before each use. Wiping was done with an unused and clean lint free cloth. All panels were then grit blasted using virgin, 180 grit Aluminum Oxide blasting media (Treibacher Scheifmettel Corporation, Niagara Falls, USA). This step was repeated until bonding surfaces of each panel are visually uniform in color appearance. At this point, the grit blasted portions of the panels were only in direct contact with other grit blasted panels or the air. A clean stream of pressurized nitrogen gas was used on each panel to remove excess grit. This was followed by wiping off any remaining grit blasting medium with a new lint free cloth. Finally, another short stream of pressurized nitrogen gas was blown over each aluminum panel.

The grit blasted aluminum lap-shear joint panels then underwent silane treatments to improve the interfacial bonding. The silane treatment consisted of 99 weight percent (90:10 ethanol:water mixture) and 1 weight percent of 3-glycidoxypropyltrimethoxysilane (GPS, Fluka) for the epoxy adhesive. Hydrolysis of the GPS was initiated by` adjusting the pH to 4.5 with acetic acid. For the polyureas adhesive, the silane treatment consisted of 99 weight percent (90:10 ethanol:water mixture) and 1 weight percent of 3-aminopropyltrimethoxysilane (APS, Fluka). APS was

allowed to self-hydrolyze in ethanol:water solution. The aluminum panels were dipped coated in their respective silane solutions for two minutes. After treatment, the aluminum panels were dried in a nitrogen stream to produce a thin, uniform coating and placed in an oven at 100 °C for 1 hour to allow for full condensation and crosslinking of the siloxane groups to the surface oxide of the aluminum panels.

Sulfo-Ferric Etch (P2)

The aluminum lap shear coupons were wiped clean with acetone and a clean paper towel. The coupons were cleaned by immersion in a 60 °C alkaline cleaning solution (Zep, Inc. Heavy Duty Alkaline Cleaner). The coupons were allowed to soak for 10 minutes then removed and rinsed with room temperature DI water for several minutes. After rinsing and while still wet, the area to be etched is immersed in the P2 etching solution at 60 °C for 20 minutes. After etching the coupon was rinsed using DI water at room temperature for 5 minutes. The coupons were air dried at room temperature for 1 hour then stored in a clean, dry environment and bonded within 16 hours of etching. The aqueous 1 liter P2 solution was prepared using 122.5 grams $Fe_2(SO_4)^3 \cdot 4H_2O$ and 0.185 liters of concentrated sulfuric acid.[14,15]

2.3 Epoxy Adhesive

The epoxy adhesive used was FM 94K Modified Epoxy Film (Cytec Engineered Materials, Inc., Havre de Grace, MD). The FM 94K film adhesive was removed from cold temperature storage and allowed to equilibrate to room temperature for approximately 30 minutes prior to layup. For out-of-autoclave processing, 6 plies of adhesive were stacked and used for bonding the single-lap-joints. The single-lap-joint tooling fixture was then vacuum bagged and heated in an oven at a rate of 2 °C/minute to an equilibration temperature of 120 °C. Cure temperature was monitored using thermocouples placed in the oven and within the lap-shear joint tooling fixture. The single-lap- joint tooling fixture required approximately 4 hours to reach 120 °C. Total cure cycle time was 6 hours, after which the oven was simply turned off and the lap-shear joint tooling fixture was allowed to slowly equilibrate to room temperature to minimize residual stress. Final bond thickness was 0.813 mm ± 0.002 mm. It is also noted that the ARL FM 94K stock was past the manufacturer's recommended shelf life and the cumulative time out of refrigerated storage was also unknown. The adhesive appeared to function properly based on observations of handling and cure behavior.

For the autoclave processed joints a single layer of FM 94K adhesive was used for bonding the joints. The single-lap-joint tooling fixture was then vacuum bagged. The autoclave (ASC Process Systems, 1.5 m x 2.1 m Econoclave) was heated at a rate of 1.5 °C/min to 121 °C, for a soak time of 2 hours. Cure temperature and pressures were monitored. The single-lap-joint tooling fixture required approximately 4 hours to reach 121°C. Total cure cycle time was 6 hours, after which the autoclave cooled at a rate of 1 °C/min to minimize residual stresses in the bonded joints. Final bond thickness was approximately 0.060 mm.

2.4 Polyurea Adhesive

The polyurea adhesive used was Versalink System C100 (Air Products and Chemicals, Inc., Allentown, PA). The C100 adhesive was hand mixed using a mix ratio of 4.5 wt Part A (resin):

1 wt Part B (curative), for approximately 2 minutes. The mixed adhesive was then de-gassed for approximately 12 minutes at room temperature under vacuum to reduce voiding during cure. The adhesive was then immediately applied to the single-lap-joints using hand held pipets, as the gel time is approximately 20 minutes. Cure at room temperature for 24 hours, followed by post curing for 3 hours at a temperature of 60 °C. Final bond thickness was 0.813 mm ± 0.002 mm.

2.5 Meta Data

It was decided that a work flow scheme which captured metadata and data at their source would best preserve data integrity. Prior to testing, each lab-shear sample was given a MSAT generated unique specimen identification (ID). The work flow scheme used here, as shown schematically in Figure 3, transfers and converts relevant load versus displacement raw data directly to MSAT as a verifiable digital asset. Adhesive materials ID and test metadata (date of preparation, specimen lot identification (ID) information, basis testing standard, surface treatment, sample preparation procedure, calibration, operator, contact information of test lab, and perceived data of test engineer's observations) are captured in the test frame software prior to testing the lap shear sample. This metadata is exported directly as a text file, whose standardized format allows for automated upload into the adhesive database. Both the metadata file and the experimental test data file are tagged with the unique specimen ID to ensure proper data affiliation in the database.

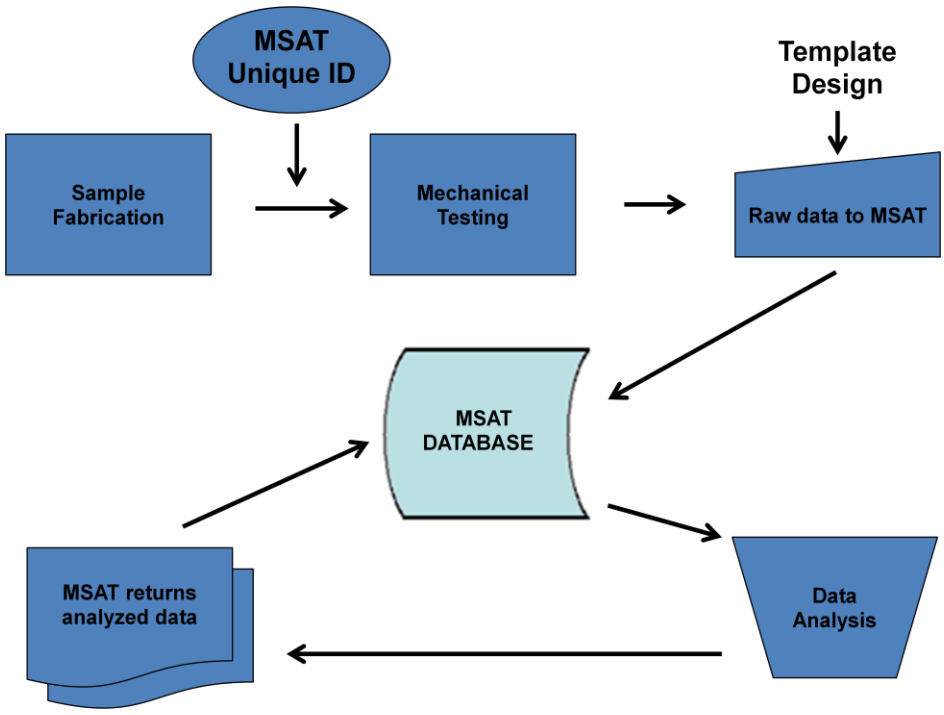

Figure 3. Work flow scheme used to transfers and converts relevant load versus displacement raw data directly to MSAT.

The mode of failure must also be considered along with the strength. Observing the failure surfaces provides insight into physical mechanism: adhesive, cohesive, or substrate failure. It was determined that image acquisition of the failure surfaces would be accomplished via a digital scanner, as they are widely available at high resolutions at reasonable expense. Considerations into file type, resolution, depth of field, and labeling, although mundane in nature, are important to ensure the files are accessible and useful for future digital image processing. Appropriate ARL "Branding", unique sample ID, and calibration scales were captured simultaneously to prevent unrecoverable accidental corruption or distortion of images.

2.6 Analysis

Standardized calculations of the maximum lap shear strength (LSS) are performed by dividing the maximum failure load (P_{max}) by the surface area of the adhesive bond (A), as shown in the Equation 1.

$$LSS = \frac{P_{\max}}{A} \quad [1]$$

This simplified first-screening analysis approach is perfectly acceptable for adhesives with reasonably linear-elastic load versus displacement response. However, the typical loads versus displacement responses for single-lap-joints bonded with damage tolerant adhesives are typically non-linear. Complex x-y plots are commonly fitted using the Levenberg-Marquardt algorithm (LMA), as described in Equation 2.[16,17]

$$S(\beta) = \sum_{i=1}^{m} \left[y_i - f(x_i, \beta) \right]^2 \quad [2]$$

The LMA is an iterative method that relies upon an initial guess for the parameter vector (β). The LMA is reported as a standard fitting routine built into Mathematica, MATLAB, and Origin.[18,19,20,21]

Despite the widespread acceptance and availability of the commercial software needed to perform the LMA, it remains desirable to further simplify the analysis of complex load versus displacement curves a commonplace computer program. Excel[22] is universally familiar at the most basic levels of mathematical analysis, but would be burdensome for manually programming the LMA. However, building an Excel analysis protocol is attainable using an adaptation of Christensen's assumption that yield can be defined as follows.[23]

$$\frac{d^3\sigma}{d\varepsilon^3} = 0, \text{ at yield} \quad [3]$$

Christensen's definition of yield stress is easily solved from basic polynomial fits of load versus displacement plots in Excel. Additionally, the resolution of the raw data typically exported from single-lap-joint testing has adequate resolution to allow for simple Riemann sum (RS) calculations for area under the curve.

$$RS = \sum_{i=1}^{n} f(x_{i-1})(x_i - x_{i-1}) \quad [4]$$

These simplifying assumptions for yield and area under the curve were used to program an Excel-based spreadsheet for single-lap-joint analysis. The spreadsheet was also made compatible for direct input into the MSAT database by leveraging the platform support from NASA. By adjusting the spreadsheet protocol for compatibility with the baseline GRANTA MI™ [24] software package, MSAT resulted in the benefit of significantly decreasing the manual operator time required for analysis to less than 5 minutes per sample. The rapid analysis protocol is ideally suited for coupling with the increasing population of adhesive samples in ARL's MSAT database to allow for data mining of relevant properties that are desirable for Army applications.

3. RESULTS

The single-lap-joint load versus displacement plots (Figure 4), failure surface (Figure 5), and results (Table 1) clearly show the wide variability possible from adhesive testing. The ARL lap shear strength results for the FM 94K adhesive (25.7 – 33.3 MPa) were all well below the manufacturer reported value of 46.6 MPa for a multitude of reasons. The ARL FM 94K was past the expiration date, was processed using different surface pretreatments, had varied bond thicknesses, and in the case of sample 20090020 bonded using out-of-autoclave vacuum bag pressure. Similar variability in adhesive/cohesive modes can also be seen in the failure surfaces of the FM 94K samples, particularly in the case of 20090020 for the lower bonding pressure and increased bond thickness. The ARL FM 94K bonded joints failed between 1.42 and 2.54 mm of crosshead extension, as measured directly by the instrument. It is unknown what the experimental load versus displacement curve for the manufacturer's reported lap shear strength of 46.6 MPa, therefore the load is represented as a constant 15000 N as derived from Equation 1.

The maximum lap shear strength for the C100 adhesive sample (20090110) was 12.0 MPa. The C100 sample failed at 3.92 mm of crosshead displacement. The total area under the load versus displacement curve is 12600 N mm, where the FM 94K samples varied from 7420 to 15200 N mm. From Table 1 the C100 samples also showed a seemingly excessively high standard deviation for the extension and area under the load versus displacement curve to maximum load. This is purely an experimental phenomenon of the sample response; as the C100 load versus displacement curves showed two peak maximums. Both of these peak maximums where nearly equivalent in height, which resulted in a roughly even distribution of maximum loads in the first and second peaks.

The apparent yield point analysis of the single-lap-joint is open for interpretation, as the value may or may not have a physical meaning for such a complex loading environment. The yield point analysis was also fairly subjective to the displacement bounds used to fit the experimental data, which is in need of a more extensive sensitivity analysis. However, the complexity of the ground vehicle bonding conditions and the subsequent non-linear adhesive properties that result in improved damage tolerance certainly require a metric to screen the degree to non-linearity.

The proposed derivative based determination of yield point is mathematically simple and easy to apply to experimental data, which warrants further investigation.

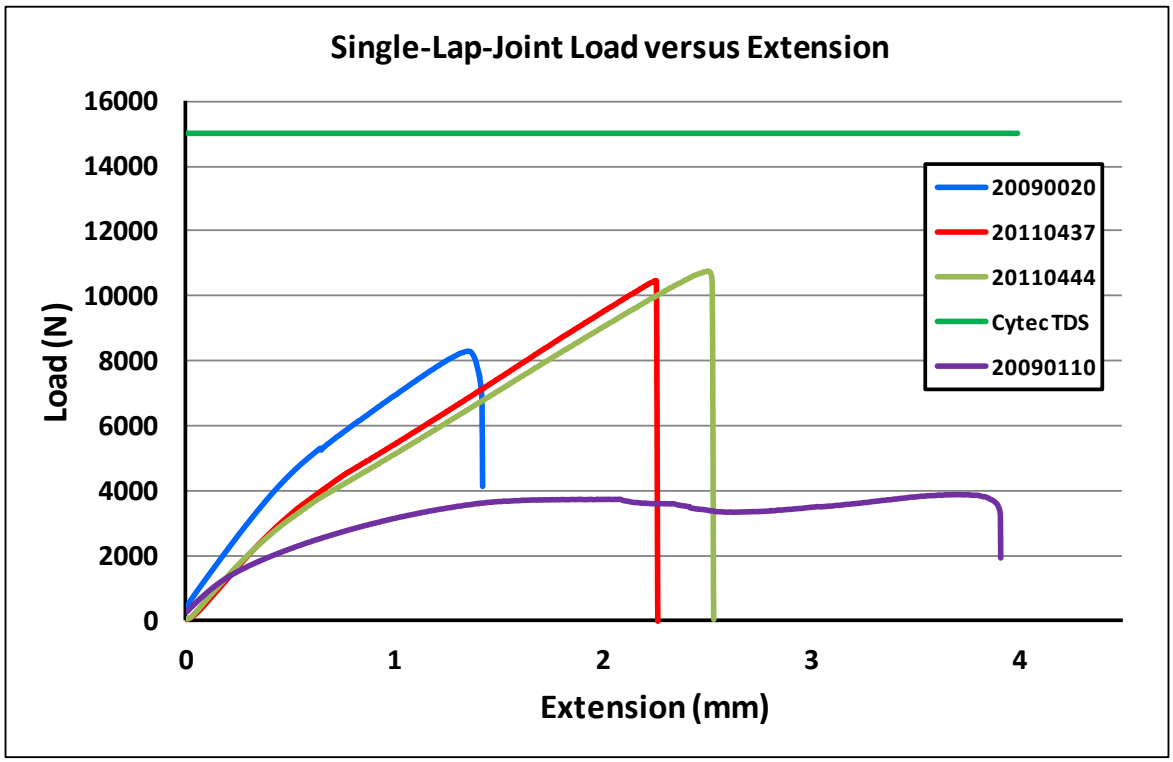

Figure 4. Single-lap-joint load versus displacement plots for samples 20090020 (FM 94K, silane pretreat, out-of-autoclave process), 20110437 (FM 94K, silane pretreat, autoclave process), 20110444 (FM 94K, P2 etch pretreat, autoclave process), and 20090110 (C100, silane pretreat, out-of-autoclave process). The FM 94K manufacturer results are shown as a constant load based on a reported single-lap-joint strength of 46.6 MPa.[25]

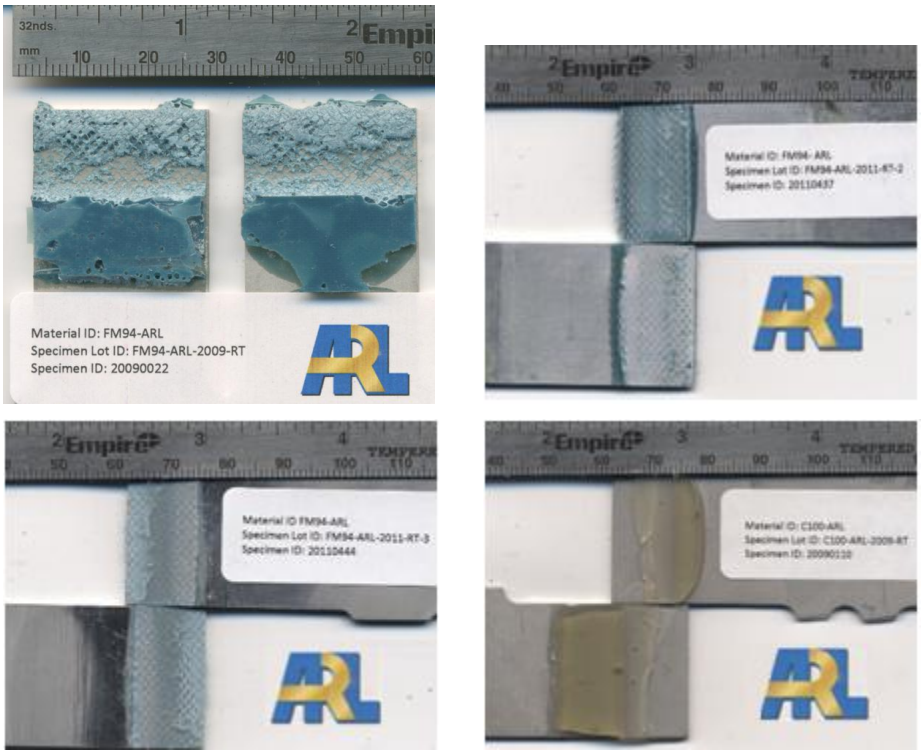

Figure 5. Single-lap-joint failure surfaces for samples 20090020 (FM 94K, silane pretreat, out-of-autoclave process, upper left), 20110437 (FM 94K, silane pretreat, autoclave process, upper right), 20110444 (FM 94K, P2 etch pretreat, autoclave process, lower left), and 20090110 (C100, silane pretreat, out-of-autoclave process, lower right).

Table 1. Single-lap-joint results for FM 94K and C100 samples plotted in Figure 4. Sample set averages and standard deviations are reported in the parenthesis.

	20090020	**20090437**	**20090444**	**Cytec TDS**	**20090110**
Adhesive	FM 94K	FM 94K	FM 94K	FM 94K	C100
Surface Preparation	Silane	Silane	P2	FLP/PAA	Silane
Cure Temperature (°C)	121	121	121	120	60
Bonding Pressure	Out-of-autoclave	Autoclave	Autoclave	Autoclave	Out-of-autoclave
Bond Thickness (mm)	0.813 ± 0.002	0.060	0.060		0.813 ± 0.002
Max Strength (MPa)	25.7 (26.8 ± 1.1)	32.4 (32.7 ± 1.6)	33.3 (33.0 ± 1.4)	46.6	12.0 (26.8 ± 1.1)
Extension at Max Load (mm)	1.35 (1.43 ± 0.08)	2.26 (2.33 ± 0.15)	2.51 (2.52 ± 0.13)		3.71 (2.79± 1.19)
Area Under the Curve to Max Load (N mm)	6910 (7500 ± 630)	13000 (13500 ± 1400)	15000 (14800 ± 1460)		11800 (8500 ± 4200)
Yield Strength (MPa)	13.3 (11.3 ± 1.4)	9.4 (8.6 ± 0.8)	7.6 (7.0 ± 0.7)		8.5 (8.2 ± 0.4)
Extension at Yield (mm)	0.45 (0.38 ± 0.06)	0.45 (0.42 ± 0.04)	0.36 (0.34 ± 0.03)		0.76 (0.65 ± 0.08)
Area Under the Curve to Yield (N mm)	1090 (800 ± 220)	660 (570± 100)	430 (380 ± 60)		1370 (1100 ± 170)
Extension at Failure (mm)	1.42 (1.50 ± 0.08)	2.27 (2.34 ± 0.15)	2.54 (2.54 ± 0.14)		3.92 (3.93 ± 0.017)
Area Under the Curve at Failure (N mm)	7420 (8060 ± 700)	13100 (13600 ± 1400)	15200 (15100± 1500)		12600 (12200 ± 300)

4. DISCUSSION

The single-lap-joint results for the FM 94K and C100 adhesives obtained by ARL are deceptive if interpreted strictly from the guidance provided by ASTM D1002. While the C100 shows the "lowest" quasi-static lap shear strength at 12.0 MPa, empirically its damage tolerance performance under high loading rate conditions appears to be significantly improved when compared to FM 94K. The ARL use of expired FM 94K adhesive and surface pretreatments not claimed by the manufacturer for testing is also deceptive when approached from a rigorous aerospace perspective. The ARL FM 94K appeared to process properly and the damage modes observed during qualitative high loading rate testing also characterized the adhesive performance as adequate. In other words, depending on the bonding configuration, the ARL FM 94K lap shear strength of 25.7 MPa is probably above a minimum threshold needed to drive specific global failure mechanisms in certain configurations bonded for high loading rate performance.

While ASTM D1002 is a very useful screening test for adhesives, the ARL single-lap-joint test results also suggest the limitations of the reporting requirements for ASTM D1002 when shifted away from an aerospace emphasis towards ground vehicle applications where concurrent strength and damage tolerance is required. Based strictly on the simple calculation of lap shear strength based on Equation 1 the C100 adhesive could be judged inferior to FM 94K. However, examining the complete load versus displacement curves and accounting for area under the curve and extension at failure provides much more insight into screening for potential damage tolerance properties. Thus, the simple reporting of peak lap shear strength without revealing the complete load versus displacement behavior is inadequate when judging potential adhesive behavior where increased damage tolerance is required. Furthermore, the bond properties need to matched to the specific high loading rate application, which further emphasizes the need to fully capture experimental and metadata for a very broad range of adhesives.

These results also lead directly to the need for increased efficiency in data collection during the transference process from experimental testing results to digital format. As can be implied from these specific results for FM 94K and C100, it can be inferred that the potential processing window for the broad range of adhesives suggested by Figure 1 will also be equally broad. If it can be shown that FM 94K will provide four different properties when processed by four different techniques, one can only imagine how complex tracking potentially hundreds of adhesives will be, which emphasized the need to insure high data pedigree during testing. From experience, it seems that the reliance on data pedigree decreases rapidly as the time difference between experimental testing and documentation increases. The significant shift in the MSAT database informatics approach that ensures a very high pedigree level is collecting the sample metadata PRIOR to testing, rather than collecting scattered sample and conditioning information after the test. The ARL results also suggest that the experimental methodology of the single-lap-joint test is useful, but that the analysis needs to provide more depth.

5. CONCLUSIONS

The purpose of this project is to support the eventual creation of a military performance standard for possible adhesive candidates for Army applications. The standard will be based on a

standardized single-lap-joint, but will call for more in-depth analysis and reporting of complete load versus displacement curves when compared to ASTM D1002, as adhesives used for Army applications have complex and non-linear behaviors. By having standardized and simple quasi-static tests the screening process is more reliable as all new data can be trusted because the majority of the variance has been eliminated. Additionally, a high level of confidence in the pedigree of the data is also ensured. Using a database to store the data is important, especially as the Army looks to industry and academia to assist in the development process of new materials to meet mission objectives. The current MSAT database allows for raw data from experimental testing to be uploaded with almost no lag time through carefully designed and preplanned automated templates.

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